

(12) UK Patent Application (19) GB (11) 2 038 819 A

(21) Application No 7942016
(22) Date of filing 5 Dec 1979
(30) Priority data
(31) 7849645
(32) 21 Dec 1978
(33) United Kingdom (GB)
(43) Application published
30 Jul 1980
(51) INT CL³
C07C 79/28 C06B 41/02 // 49/00
(52) Domestic classification
C2C 220 227 22Y 292
29Y 30Y 332 365 36Y 631
633 661 662 80X AA MK
C1D 6A1B 6A1D 6A1F
6A1G 6A2C 6A2N 6B3
(56) Documents cited
None
(58) Field of search
C2C
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(54) Initiatory Explosives
Comprising a Lead Salt of 3,5-
Dinitro-catechol

(57) An initiatory explosive comprises the anhydrous normal lead (II) salt of 3,5 dinitro catechol (3,5-dinitro 1,2 dihydroxy benzene), which may be used alone or form part of an explosive composition mixed for example with oxidants, sensitizers etc. The explosive may be conveniently prepared by the reaction between dinitrocatechol and a suitable lead

compound, or by double decomposition, in aqueous media, and is suited for production in situ in an explosive device. It has a mechanical sensitivity as good as conventional materials such as lead styphnate or resorcinat, but has a considerably lower electrostatic sensitivity, which leads to safer handling. The explosive and compositions containing it are suitable for use in most initiatory explosive devices such as percussion caps and stab sensitive detonators as a simple substitute for conventional materials.

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SPECIFICATION **Initiatory Explosives**

This invention relates to metal salts of nitrated phenols which have explosive properties and may be used as initiatory explosives. Usually lead salts of nitrated phenols, especially poly-nitrated phenols such as 2,4-dinitroresorcinol, 4,6-dinitroresorcinol and 2,4,6-trinitroresorcinol (styphnic acid) have found extensive use as primary explosives.

In particular, both normal and monobasic lead styphnates are in general use in initiatory explosive compositions for example in priming compositions in percussion caps. In such applications the primary explosive is mixed with an oxidant such as a metal nitrate, chlorate or perchlorate, a sensitizer usually tetrazene and usually one or more other ingredients such as lead dioxide, antimony sulphide, calcium silicide, aluminium and ground glass. Similarly lead 2,4-dinitroresorcinate is in general use in initiatory explosive compositions for example in composite detonators, mixed with an oxidant such as barium nitrate or potassium perchlorate, a sensitizer such as tetrazene and sometimes other additives such as aluminium or lead dioxide to provide an explosive composition sensitive to initiation by stabbing.

In addition to mechanical sensitiveness lead dinitroresorcinate and especially lead styphnate are also sensitive to electrostatic charges and stringent safety procedures are required in the manufacture and handling of these explosives and derived compositions. Hazards in bulk preparation of these compositions may be considerably reduced if an explosive may be used possessing less electrostatic sensitiveness while maintaining the required mechanical sensitiveness. Alternatively the hazard may be reduced by preparing the sensitive explosive ingredient in situ in the required composite mixture thus eliminating the need for prior manufacture in bulk of the sensitive explosive composition. Such a method for lead styphnate is claimed in Br. Pat. 1,479,569 but the efficacy of the method depends on the purity of the styphnic acid used.

According to this invention, an initiatory explosive comprises the anhydrous normal lead (II) salt of 3,5-dinitrocatechol (3,5-dinitro-1,2-dihydroxybenzene) hereinafter termed lead DNC. The lead DNC may be used alone as a single component initiatory explosive but will normally form part of an explosive composition containing other ingredients of the types aforementioned, for example, in lead styphnate compositions. 3,5-dinitrocatechol (DNC) may be prepared by for example nitration of catechol as described in *Chemische Berichte* 26 (1893) p. 2183.

The lead (II) salt may then be prepared either by direct reaction of the DNC with a suitable lead compound, such as the oxide, carbonate, formate or acetate, in the presence of moisture or, more conventionally, via a soluble DNC salt such as the sodium, potassium or magnesium salt by reaction with a soluble lead salt such as lead nitrate or lead acetate in aqueous solution.

According to a preferred aspect of the invention lead DNC is produced in situ in a detonator or cap by filling the detonator or cap with a dry mixture comprising at least one precursor of the lead DNC and any other ingredients required in the final composition and subsequently adding an aqueous liquid to cause formation of the lead DNC. Thus the dry filling composition may contain DNC and lead oxide and the aqueous liquid is simply water. Alternatively the filling mixture may contain DNC but no lead compound when the aqueous liquid may be a solution or suspension of a lead salt, preferably lead acetate. The detonator or cap is then heated to complete the reaction, if necessary, and to drive off the water and, in the case of using lead acetate, the acetic acid produced as a by product of the reaction.

In another aspect of the invention premixes of the above types may be prepared in much larger quantities with less hazard than for example lead styphnate compositions and converted in bulk by the aforescribed methods to the more sensitive explosive composition suitable for multiple filling of detonators and caps. This method presents a simpler and less hazardous alternative to an existing type of process in which lead styphnate is prepared and handled in the wet state, then remotely dried and mixed with the other ingredients of the final composition. An advantage of the invention is that the normal drying cycle as used for wet lead styphnate is sufficient to complete the reaction between DNC and the lead compound, and dry the product.

The lead DNC produced by the processes described above shows similar mechanical sensitiveness to lead styphnate (F of I (Rotter Test)=20) but considerably less electrostatic sensitiveness than lead styphnate both as a single explosive and in mixtures with other ingredients as mentioned previously.

Lead DNC is suitable for use in composite detonators using for example mixtures of lead dinitroresorcinate, barium nitrate, tetrazene (L composition) or variants thereof as a stab sensitive increment, and in percussion caps using lead styphnate compositions such as VH2 (lead styphnate, barium nitrate, tetrazene, lead dioxide, antimony sulphide, calcium silicide) as a direct replacement for the lead DNC or the lead styphnate respectively. Lead DNC compositions of the above types are less violent than equivalent lead styphnate compositions but a more vigorous output may be obtained by substituting the barium nitrate oxidant by potassium chlorate or potassium perchlorate. Thus a composition based on lead DNC, potassium perchlorate, tetrazene and, if necessary, a small amount of aluminium or other finely divided additive to aid mixing and filling operations, may be considered as a substitute for standard VH2 compositions.

The preparation of lead DNC using the methods outlined above will now be described by way of example.

Example 1

To a stirred suspension of 2 g 3.5 DNC in 50 ml water at 20°C was added 2.23 g lead monoxide. The mixture was heated to 60°C and stirred for 30 minutes before cooling to 35°C. The product was allowed to settle for 2 minutes, the supernatant liquid decanted, and the product filtered and washed with water followed by methylated spirit. The product was then cold dried by drawing air through it or hot dried at about 50°C.

Yield: 100% Theoretical

Example 2

As a model for in situ production of lead DNC a mixture of 2 g 3.5 DNC and 2.23 g lead monoxide was made into a paste with 1.25 ml water and dried on a hot plate at about 50°C. The resultant product was lead DNC.

Yield: 100% Theoretical

Example 3

To a stirred suspension of 2 g 3.5 DNC in 50 ml water at 20°C was added 12 ml of 320 g/l lead acetate trihydrate solution. The mixture was heated to 90°C, stirred for 15 minutes at 90°C and cooled to 35°C. the product was then treated as in Example 1.

Yield: 100% Theoretical

Example 4

As an alternative to the in situ process of Example 1 a mixture of 2 g 3.5 DNC and 3.79 g lead acetate trihydrate was moistened with 1—2 ml water, and dried at about 50°C to give lead DNC.

Yield: 100% Theoretical

Example 5

A saturated solution of lead acetate trihydrate containing 3.79 g lead acetate trihydrate was added to 2 g 3.5 DNC. The paste was dried at about 50°C to yield lead DNC.

Yield: 100% Theoretical

Example 6

To 3 litres of aqueous magnesium 3,5-dinitrocatechate containing 30 g/l 3.5 DNC and 3.75 g/l magnesium oxide stirred at 65°C was added 0.541 aqueous lead acetate, containing 3.15 g/l lead acetate trihydrate, during 20 minutes. The mixture was stirred for a further 5 minutes at 65°C before cooling to 35°. The product was then treated as in Example 1.

Yield: 85% Theoretical

Typical sensitiveness properties of lead DNC prepared according to the invention are given below by way of example, and are compared with the corresponding properties of two grades of lead styphnate: A which is commercial normal lead styphnate, and B which is normal lead styphnate modified to give a reduced electrostatic sensitivity, as currently used.

SCC Test No*	Test	Lead DNC	Lead B	Styphnate A
40	1/72 Impact; Fof I (RDX=80)	20	20	20
	2/72 Friction (Mallet)			
	Boxwood on Yorkstone/Hardwood/Softwood.		all 100/100/100	
45	13/66 Emery Friction	6	8	7
	Velocity (ft sec) for 50% ignition			
	3/66 Temperature of Ignition (°C)	303	257	254
	6/66 Electrostatic			
	(1) Metal/Metal electrodes			
50	Minimum Energy	50 μ J	25 μ J	15 μ J
	Minimum Capacitance	(520 pF)	(520 pF)	(520 pF)
	(2) Rubber/Metal electrodes			
	Minimum Energy	3800 μ J	15 μ J	0.3 μ J
	Minimum Capacitance	255 pF	25 pF	<10 pF

*SCC Tests as described in UK Sensitiveness Collaboration Committee (SCC) Explosive Hazard Assessment, Manual of Tests No. 3.

Claims

1. An initiatory explosive comprising the anhydrous normal lead (II) salt of 3,5-dinitrocatechol.
2. An initiatory explosive according to claim 1 which is stab sensitive.
3. An initiatory explosive according to claims 1 and 2 also containing barium nitrate and tetrazene.

4. An initiatory explosive according to claim 3 also containing lead dioxide, antimony sulphide and calcium silicide.
5. An initiatory explosive according to claims 1 and 2 also containing potassium perchlorate and tetrazene.
- 5 6. An explosive device containing an initiatory explosive according to any one of claims 1 to 5.
7. An explosive device according to claim 6 which is mechanically fired. 5
8. A process for the production of an explosive device according to claim 5 or 6, wherein the said lead salt of dinitrocatechol is produced in situ in an explosive device.
9. A process according to claim 8 in which the said lead salt of dinitrocatechol is produced by introducing a dry mixture comprising at least one precursor of said lead salt and any other ingredients required in the final composition into the explosive device, adding an aqueous liquid to form the lead salt, heating if necessary to complete the reaction and finally driving off remaining water and any volatile side products. 10
10. A process according to claim 9 in which the dry mixture contains lead oxide or a lead salt of a volatile acid and dinitrocatechol, and the aqueous liquid comprises water. 15
11. A process according to claim 9 in which the dry mixture contains dinitrocatechol, and the aqueous liquid comprises a solution of a lead salt of a volatile acid.
12. A process according to claims 10 and 11 in which the lead salt is lead acetate.
13. An explosive device prepared by a process according to any of claims 8 to 12.

Printed for Her Majesty's Stationery Office by the Courier Press, Leamington Spa, 1980. Published by the Patent Office,
25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.